

California Environmental Protection Agency



# **Air Resources Board**

**PROCEDURE FOR DETERMINING THE COMPOSITION OF ELEMENTS IN  
PARTICULATE MATTER FROM MOTOR VEHICLE EXHAUST BY ENERGY  
DISPERSIVE X-RAY FLUORESCENCE (EDXRF) SPECTROSCOPY**

**Standard Operating Procedure MV-Aerosol 156  
Version 2.0**

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Aerosol Analysis and Methods Evaluation Section  
Chemical Analysis & Emissions Research Branch  
Emissions Compliance, Automotive Regulation and Science  
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# SOP No. 156 COMPOSITION OF ELEMENTS BY ENERGY DISPERSIVE X RAY FLUORESCENCE (EDXRF) SPECTROSCOPY

## 1. Introduction

This document describes a non-destructive procedure using the Energy Dispersive X-Ray Fluorescence (EDXRF) spectroscopy to determine the composition of elements in particulate matter collected on Polytetrafluoroethylene (PTFE) membrane filters. When samples are irradiated by X-rays within the instrument sampling chamber, they emit X-ray fluorescence that is characteristic for each element. The intensity of emitted fluorescence energy is proportional to the surface concentration of each element (in units of  $\mu\text{g}/\text{cm}^2$ ) and is used for their quantification.

The background X-ray spectrum of a blank filter is subtracted from that of the sample spectrum. The net intensity is proportional to the concentration of the corresponding element collected on the sample filter.

## 2. Interferences and Limitations

- 2.1. This procedure is capable of quantitative analysis of the non-volatile elements with atomic numbers 11 (sodium) through 92 (uranium). The 44 target elements that are measured in vehicle exhaust samples are listed in Table 1. The vacuum condition in EDXRF during sample analysis causes some loss of elements associated with volatile and semi-volatile compounds. For those elements, the accurate quantification cannot be achieved.
- 2.2. EDXRF method does not distinguish oxidation states; therefore only total elemental concentrations are quantified.
- 2.3. EDXRF spectral interferences cannot be avoided. The Epsilon 5 system uses secondary polarizing targets to minimize the effect of spectral overlapping.
- 2.4. Particulate matter is assumed to be deposited uniformly on the filter. The elements in the particulate matter which are not uniformly deposited over the whole collection area cannot be accurately quantified.
- 2.5. The procedure only applies to PTFE filters. Different type of filter media may exhibit severe background interference.

## 3. Safety

- 3.1. EDXRF instrument generates X-rays. The instrument has a built-in radiation shielding and safety interlock system with X-ray safety certifications. A Geiger counter is used to monitor for any radiation leaks.

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- 3.2. The EDXRF instrument uses liquid nitrogen to keep the X-ray detector at low temperature. The instrument's liquid nitrogen reservoir is refilled at least once a week. Liquid nitrogen is a cryogenic hazard and causes severe burns on contact with skin, eyes, or lungs. Wear protective gear (cryogenic gloves, safety glasses, lab coat) when handling liquid nitrogen (Reference: Occupational Safety and Health Administration).
- 3.3. Liquid nitrogen vapor displaces air while boiling off the liquid, and is an asphyxiation hazard. Keep the laboratory doors open when filling the liquid nitrogen reservoir.
- 3.4. For general laboratory safety procedures, consult the Chemical Hygiene Plan. Safety Data Sheets (SDS) are available in the laboratory.

### **4. Equipment and Supplies**

- 4.1. The PANalytical Epsilon 5 High Performance EDXRF Spectrometer is used for the quantitative analysis of elements. This consists of the following instrument components:
  - 4.1.1 X-ray Tube: A scandium/tungsten (Sc/W) dual anode X-ray tube, with gadolinium (Gd) side window, with a water cooling system.
  - 4.1.2 HV Generator: 600 Watt maximum power, output voltage range 25-100 kV adjustable with steps of 1 kV, output mA range 0.5-24 mA adjustable by 0.1 mA.
  - 4.1.3 A 3-dimensional polarized optical path with nine automated polarizing secondary targets: These polarizing secondary targets and their corresponding measuring conditions are used for exciting target elements are listed in Table 2.
  - 4.1.4 X-ray Detector: A germanium (Ge) detector with beryllium (Be) window cooled by liquid nitrogen.
  - 4.1.5 Sample chamber equipped with automated controls: The chamber is in a vacuum environment during sample analysis.
  - 4.1.6 Computer system running on Windows 7 with PANalytical Epsilon 5 application software version 2.0N / ICSW 2.11.
- 4.2 Laboratory supplies and other Equipment:
  - 4.2.1 Step up transformer.
  - 4.2.2 Liquid nitrogen source with a pressure regulator at 0-50 psi.

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- 4.2.3 Cryogenic gloves, lab coat, safety glasses.
- 4.2.4 Latex gloves, disposable, class 100, powder free.
- 4.2.5 Stainless steel forceps with flat wide tips for transferring filters.
- 4.2.6 Solid sample holders (part #9430 050 01221) and sample holder inserts, for 50 mm filters (part #9430 050 11501)
- 4.2.7 Polycarbonate film, 6 microns thickness

### **5. Standards**

#### 5.1. Elemental Calibration standards:

- 5.1.1 These standards are thin vapor-deposited films of ultra-pure non-interfering elemental materials deposited on Nuclepore<sup>®</sup> polycarbonate aerosol membrane.
- 5.1.2 The calibration standard concentrations of the target elements are listed in Table 1.
- 5.1.3 Standards can be purchased from Micromatter, Inc. (4004 Westbrook Mall, Vancouver, BC, V6T 2A3) or from nanoXRF UHV Technologies, Inc. (450 South Freeway, Fort Worth, Texas 76104-3503).

#### 5.2. Drift correction standard: FLX S13 (22/07/2011, 10, FLUXANA #D Elektronik).

### **6. Drift Correction**

The X-ray tube and detector can degrade, and the response of the other components can also change over time. The drift correction is conducted once a month using the drift correction standard, FLX S13.

### **7. Calibration**

- 7.1 The target elements are calibrated under their usual measurement conditions and utilize their usual secondary targets. Each element's measurement conditions and corresponding secondary target are listed in Table 2.

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- 7.2 Calibration of all the elements is typically valid for one year.
- 7.3 Recalibration of any elements may need to be performed if the quality control result of a particular element exceeds the limits, when the X-ray tube or the detector is replaced, or when there is a repair that can affect the calibration.

### **8. Sample Handling and Analysis**

#### **8.1 Sample Handling:**

- 8.1.1 Sample filters are submitted to the laboratory and stored in the freezer until analyzed.
- 8.1.2 Filter samples are manually inspected before analysis. If irregularities are found, a senior staff shall be consulted. Any irregularities are recorded in the logbook.

#### **8.2 Sample Analysis:**

Two methods are used for EDXRF analysis. The primary method is used for the majority of analyses, including all gasoline vehicle exhaust samples. The supplemental method is intended primarily for diesel exhaust and other very high loading samples, and implements a layer of polycarbonate (PC) film over the sample to protect the instrument from possible contamination.

- 8.2.1 The analyst must be trained and familiar with the operation of the EDXRF system.
- 8.2.2 Create a sample list on the logbook and transfer the information to the "Sample Changer Measurement" window. Specify the number of repeats for each sample, and the sample rack position.
  - i. For the primary method, select the "Whatman Air Filter" measurement application.
  - ii. For the supplemental method using PC film, select the "Whatman Air Filter – polycarbonate film" measurement application.
- 8.2.3 It is important that the deposit is not scraped, smudged or smeared when loading the filters into the sample holders.

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- i. For the primary method, carefully transfer the filter with a clean forceps (with the deposit side up) to the sample holder insert. Place the sample holder cup over the filter to complete the sample assembly.



Place the sample assembly in the sample tray with the filter facing down, according to the position on the sample list. Place the tray inside the sampling chamber.

- ii. For the supplemental method using PC film, the filters are first loaded into customized filter pucks, with a sheet of polycarbonate film placed over the top of the filter surface.



With the supplemental method, the inner sample holder ring is not used.

### 8.2.4 Load sample cups into trays and place into instrument.



Primary method



Supplemental method



Instrument

- 8.3 A typical analytical sequence includes a blank filter for background correction, a QC standard at the beginning of the sample batch, a sample replicate for every 10 samples or less, and a QC standard at the end of the sample batch.
- 8.4 When the analyses are completed, load the filters back into the sample containers for storage.

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## 9. Quality Control

### 9.1 Quality Control (QC) Standard: FLX S13

9.1.1 FLX S13 is analyzed before and after each set of samples. The limits are defined as follows:

Lower Control Limit (LCL) = mean value - 3 times the standard deviation  
Lower Warning Limit (LWL) = mean value - 2 times the standard deviation  
Upper Warning Limit (UWL) = mean value + 2 times the standard deviation  
Upper Control Limit (UCL) = mean value + 3 times the standard deviation

9.1.2 The values for the control limits were calculated from the results of 20 analyses of the FLX S13, shown in Table 3.

9.1.3 QC values outside the control limit are considered as a “QC failure.” If any of the QC values fall between the control and warning limits, it is considered a QC “warning”. If the second QC value falls within the warning limits, it is considered a QC failure. QC failure requires corrective actions such as recalibration of the elements.

### 9.2 Replicate sample analysis

9.2.1 At least ten percent of the samples are randomly selected for replicate analysis. The relative percent difference (RPD) between the pair of analyses is calculated for each element above the reporting limit as follows:

$$RPD = \frac{|\text{Sample Conc.} - \text{Replicate Conc.}|}{\text{Average Conc. of Both Analyses}} \times 100$$

9.2.2 The limit on the allowable RPD is established based on the average concentration of the replicate runs, as shown in the following table:

Average Measurement for Replicate Runs	Allowable RPD (%)
1 to <3 times RL	= or <60%
3 to 5 times RL	= or <30%
Greater than 5 times RL	= or <10%

9.2.3 If the RPD of any of the target elements is greater than the allowable limit, the samples within the batch are re-analyzed.

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### 10 Reporting Limit

- 10.1 Data reporting for the elements is based on the significant figures of the calibration standards listed in Table 1.
- 10.2 The reporting limit (RL) is based on 38 blank filters analyzed from 2012 to 2014. RL is equal to the mean plus 3 times the standard deviation.
- 10.3 Sampling area of 11.34 cm<sup>2</sup> is used to convert the reporting limit from surface concentration to mass for each element for a 47mm PTFE filter. The reporting limits are listed in Table 4.
- 10.4 Any element with mass detected below the reporting limit will be reported as <RL.

### 11 Data Handling

- 11.1 Confirm that all QC criteria are met before reporting data. Otherwise, make corrections and re-analyze samples.
- 11.2 Data analysis is processed by the Epsilon 5 software.
- 11.3 Export the report generated by the Epsilon 5 software to an Excel file. Back up data periodically.
- 11.4 Raw data in µg/cm<sup>2</sup> from Epsilon 5 is transferred to the SLB LIMS, where it is converted to µg/filter (by multiplying the sample deposit area of 11.34 cm<sup>2</sup>) before reporting.

### 12 Reference standard

Standard Reference Material (SRM) 2783 from National Institute of Standards and Technology (NIST) is analyzed once a quarter. SRM 2783 is deposited on a polycarbonate membrane filter. A blank polycarbonate membrane filter (from NIST) is analyzed for background correction. Table 5 shows the NIST certified concentrations values and ARB values.

### 13 Maintenance

- 13.1 It is important that the Epsilon 5 system stays ON (Epsilon 5, computer and Epsilon 5 software). If the system is shut down for more than 2 hours, a maintenance call might be needed.

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- 13.2 The XRF detector requires routine calibration and continuous supply of liquid nitrogen. Fill the liquid nitrogen reservoir once a week and recalibrate the detector after every liquid nitrogen refill. Allow the detector temperature to stabilize for at least an hour after the liquid nitrogen refill before initiating detector calibration.
- 13.3 Check the instrument status on the software display screen and alert lights on the instrument often. If the X-ray alert lights (yellow light on top of the Epsilon 5 EDXRF, light inside of the instrument loading bay, or LEDs on the front of the instrument housing) are OFF, it indicates that the X-ray tube is not on. Check for alert messages in the software, and call for service if necessary.

### 14. Registration of the X-Ray Tube Radiation

The X-Ray radiation registration renewal is required every two years. The Radiation Machine Registration renewal invoices are issued by the California Department of Public Health Radiologic Health Branch, MS 7610, Registration Unit P.O. Box 997414 Sacramento, CA 95899-7414.

### 15. References

- 15.1 U.S. Environmental Protection Agency, June 1999. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air , Compendium Method IO-3.3, *Determination of Metals in Ambient Particulate Matter using X-ray Fluorescence (XRF) Spectroscopy*, EPA/625/R-96/010a, Office of Research and Development
- 15.2 ARB SOP MLD 034, January 2006: *Standard Operating Procedure for the Determination of Elemental Concentrations in Ambient Air by Energy-Dispersive X-ray Fluorescence (XRF) Spectroscopy*
- 15.3 Epsilon 5 software Help manual: PANalytical, Incorporated, 117 Flanders Road, Westborough, MA 01581
- 15.4 California Code of Regulations (CCR), title 17, section 30108  
<http://www.cdph.ca.gov/certlic/radquip/Pages/RadiologicEquipment.aspx>
- 15.5 Occupational Safety and Health Guidelines for Nitrogen  
<http://www.osha.gov/SLTC/healthguidelines/nitrogen/recognition.html>
- 15.6 SLB Chemical Hygiene Plan, in draft, submitted to ARB safety officer for review and approval, December 2012.

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**16. Revision Record**

<b>Revision</b>	<b>Date</b>	<b>Responsible Person</b>	<b>Description of Change</b>	<b>Status</b>
	January 2013	Luzviminda Salazar	Initial release	Version 1.0
1	August 2014	Erin Shields	Updated division and branch names	Version 1.1
2	January 2016	Erin Shields	Updated reporting limits, added supplemental method for diesel exhaust samples, and editorial changes	Version 2.0

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**17 Tables**

**Table 1 Target elements and the corresponding calibration standard concentration (Conc. uncertainty  $\pm$  5%)**

<b>Target Element 1</b>	<b>Conc. (<math>\mu\text{g}/\text{cm}^2</math>)</b>	<b>Target Element 2</b>	<b>Conc. (<math>\mu\text{g}/\text{cm}^2</math>)</b>	<b>Serial Number of Standards</b>
sodium	6.02	chlorine	9.28	34063 NaCl
magnesium	7.26	fluorine	11.3	34064 MgF <sub>2</sub>
aluminum	22.2			34065 Al metal
aluminum	51.8			12906 Al metal
silicon	11.0	oxide	6.28	34066 SiO
phosphorous	6.80	gallium	15.3	34067 GaP
sulfur	5.94	copper	11.8	34068 CuS <sub>x</sub>
potassium	6.90	chlorine	7.61	34069 KCl
calcium	8.11	fluorine	7.69	34070 CaF <sub>2</sub>
scandium	8.20	fluorine	10.4	34071 ScF <sub>3</sub>
titanium	17.7			34072 Ti metal
vanadium	20.8			34073 V metal
chromium	17.6			34074 Cr metal
manganese	18.7			34075 Mn metal
iron	14.8			34076 Fe metal
iron	49.4			12907 Fe metal
cobalt	15.7			34077 Co metal
nickel	17.0			34078 Ni metal
copper	16.9			34079 Cu metal
zinc	5.93	tellurium	11.6	34080 ZnTe
germanium	17.3			34081 Ge metal
selenium	25.6			30482 Se metal
cesium	12.2	bromine	7.36	34083 CsBr
rubidium	7.69	iodine	11.4	34084 RbI
strontium	11.9	fluorine	5.14	34085 SrF <sub>2</sub>
yttrium	13.5	fluorine	8.67	34086 YF <sub>3</sub>
molybdenum	11.5	oxide	5.74	34087 MoO <sub>3</sub>
rhodium	14.9			34088 Rh metal
palladium	17.6			34089 Pd metal
silver	19.6			34090 Ag metal
cadmium	10.7	selenium	7.55	34091 CdSe
indium	18.7			34092 In metal
tin	15.5			34093 Sn metal
antimony	16.4			34094 Sb metal
tellurium	18.0			34095 Te metal
barium	13.0	fluorine	1.80	34096 BaF <sub>2</sub>
lanthanum	15.2	fluorine	6.23	34097 LaF <sub>3</sub>
cerium	13.9	fluorine	5.67	34098 CeF <sub>3</sub>
thallium	16.4	chlorine	2.85	34099 TlCl
lead	19.5			34100 Pb metal
bismuth	16.5			34101 Bi metal
platinum	17.4			12908 Pt metal
zirconium	11.5	fluorine	9.57	12909 ZrF <sub>4</sub>

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**Table 2 Nine polarizing secondary targets, the measuring conditions and the corresponding elements of interest**

No.	Polarizing Secondary Target (Elemental composition)	Measuring Conditions				Element of interest
		kV	mA	Measured time (seconds)	Condition Name	
1	aluminum (Al)	25	24	500	Mg	Mg
2	calcium fluoride (CaF <sub>2</sub> )	40	15	600	Si-K	Al, Si, P, S, Cl, K
3	iron (Fe)	75	8	400	Ti-Cr	Ca, Sc, Ti, V, Cr
4	germanium (Ge)	75	8	400	Cu-Zn	Mn, Fe, Co, Ni, Cu, Zn
5	zirconium (Zr)	100	6	400	Rb_Re-Tl	Ga, Ge, Se, Br, Rb, Pt, Tl, Pb
6	molybdenum (Mo)	100	6	400	Sr-Y_Pb-U	Bi
7	silver (Ag)	100	6	400	Mo-Tc	Zr
8	cerium oxide (Ce <sub>2</sub> O <sub>3</sub> )	100	6	400	I	Mo, Sn, Sb, Te, I
9	Barkla target aluminum oxide (Al <sub>2</sub> O <sub>3</sub> )	100	6	500	Xe-La	Rh, Pd, Ag, Cd, In, Cs, Ba, La and Ce

**Table 3 Quality control limits for selected elements**

Element	Mean (ug/cm <sup>2</sup> )	One standard deviation (ug/cm <sup>2</sup> )	LCL (ug/cm <sup>2</sup> )	LWL (ug/cm <sup>2</sup> )	UWL (ug/cm <sup>2</sup> )	UCL (ug/cm <sup>2</sup> )
sulfur	1.28	0.03	1.19	1.22	1.34	1.37
chromium	8.77	0.05	8.62	8.67	8.87	8.92
iron	13.4	0.1	13.1	13.2	13.6	13.7
copper	27.1	0.1	26.8	26.9	27.3	27.4
lead	279	2	273	275	283	285

Lower Control Limit (LCL) = mean value - 3 times the standard deviation

Lower Warning Limit (LWL) = mean value - 2 times the standard deviation

Upper Warning Limit (UWL) = mean value + 2 times the standard deviation

Upper Control Limit (UCL) = mean value + 3 times the standard deviation

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**Table 4 Reporting limit for the elements analyzed by EDXRF**

Element	Abrv	Primary RL (ug/cm <sup>2</sup> )	Primary RL (ug/filter)	RL with film (ug/cm <sup>2</sup> )	RL with film (ug/filter)
magnesium	Mg	0.0579	0.6569	N/A	N/A
aluminum	Al	0.2382	2.7006	N/A	N/A
silicon	Si	0.0250	0.2833	0.0781	0.8852
phosphorus	P	0.0062	0.0698	0.0106	0.1205
sulfur	S	0.0172	0.1953	0.0172	0.1953
chlorine	Cl	0.1014	1.1500	0.1014	1.1500
potassium	K	0.0240	0.2723	0.0240	0.2723
calcium	Ca	0.0542	0.6143	0.0542	0.6143
scandium	Sc	0.0371	0.4213	0.0371	0.4213
titanium	Ti	0.0101	0.1149	0.0101	0.1149
vanadium	V	0.0006	0.0070	0.0006	0.0070
chromium	Cr	0.0079	0.0901	0.0079	0.0901
manganese	Mn	0.0051	0.0575	0.0051	0.0575
iron	Fe	0.0741	0.8405	0.0741	0.8405
cobalt	Co	0.0053	0.0601	0.0053	0.0601
nickel	Ni	0.0033	0.0377	0.0033	0.0377
copper	Cu	0.0042	0.0472	0.0042	0.0472
zinc	Zn	0.0366	0.4155	0.0366	0.4155
gallium	Ga	0.0053	0.0607	0.0053	0.0607
germanium	Ge	0.0070	0.0799	0.0070	0.0799
selenium	Se	0.0582	0.6598	0.0582	0.6598
bromine	Br	0.0061	0.0693	0.0061	0.0693
rubidium	Rb	0.0058	0.0654	0.0058	0.0654
strontium	Sr	0.0055	0.0619	0.0055	0.0619
yttrium	Y	0.0042	0.0472	0.0042	0.0472
molybdenum	Mo	0.0494	0.5607	0.0494	0.5607
rhodium	Rh	0.1302	1.4766	0.1302	1.4766
palladium	Pd	0.0370	0.4195	0.0370	0.4195
silver	Ag	0.0336	0.3806	0.0336	0.3806
cadmium	Cd	0.0383	0.4345	0.0383	0.4345
indium	In	0.0327	0.3707	0.0327	0.3707
tin	Sn	0.0241	0.2736	0.0241	0.2736
antimony	Sb	0.0155	0.1763	0.0155	0.1763
tellurium	Te	0.0773	0.8762	0.0773	0.8762
iodine	I	0.1068	1.2111	0.1068	1.2111
cesium	Cs	0.0779	0.8835	0.0779	0.8835
barium	Ba	0.1010	1.1451	0.1010	1.1451
lanthanum	La	0.0949	1.0757	0.0949	1.0757
cerium	Ce	0.1187	1.3459	0.1187	1.3459
thallium	Tl	0.0109	0.1231	0.0109	0.1231
lead	Pb	0.0177	0.2007	0.0177	0.2007
bismuth	Bi	0.0123	0.1400	0.0123	0.1400
platinum	Pt	0.0155	0.1762	0.0155	0.1762
zirconium	Zr	0.0081	0.0924	0.0081	0.0924

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**Table 5 Certified mass loading values of selected elements in NIST SRM 2783 for EDXRF**

Element	NIST Certified Conc. ( $\mu\text{g}/\text{cm}^2$ )	NIST Conc. 95% uncertainty ( $\mu\text{g}/\text{cm}^2$ )	ARB Primary method 7/30/2014 ( $\mu\text{g}/\text{cm}^2$ )	ARB PC film method 7/30/2014 ( $\mu\text{g}/\text{cm}^2$ )
Aluminum*	2.33	0.12	2.237	n/a
Potassium	0.53	0.03	0.47	0.47
Calcium	1.325	0.066	1.273	1.385
Titanium	0.15	0.01	0.16	0.17
Chromium	0.014	0.001	0.016	0.01
Iron	2.661	0.133	2.547	2.611
Nickel	0.007	0.0004	0.007	0.004
Copper	0.041	0.002	0.037	0.038
Zinc	0.18	0.01	0.19	0.19
Antimony**	0.0072	0.0004	<RL	<RL
Barium**	0.034	0.002	<RL	<RL
Lead	0.032	0.002	0.034	0.035

\* Aluminum is only reported with conventional method

\*\* Elements with concentrations below reporting limits